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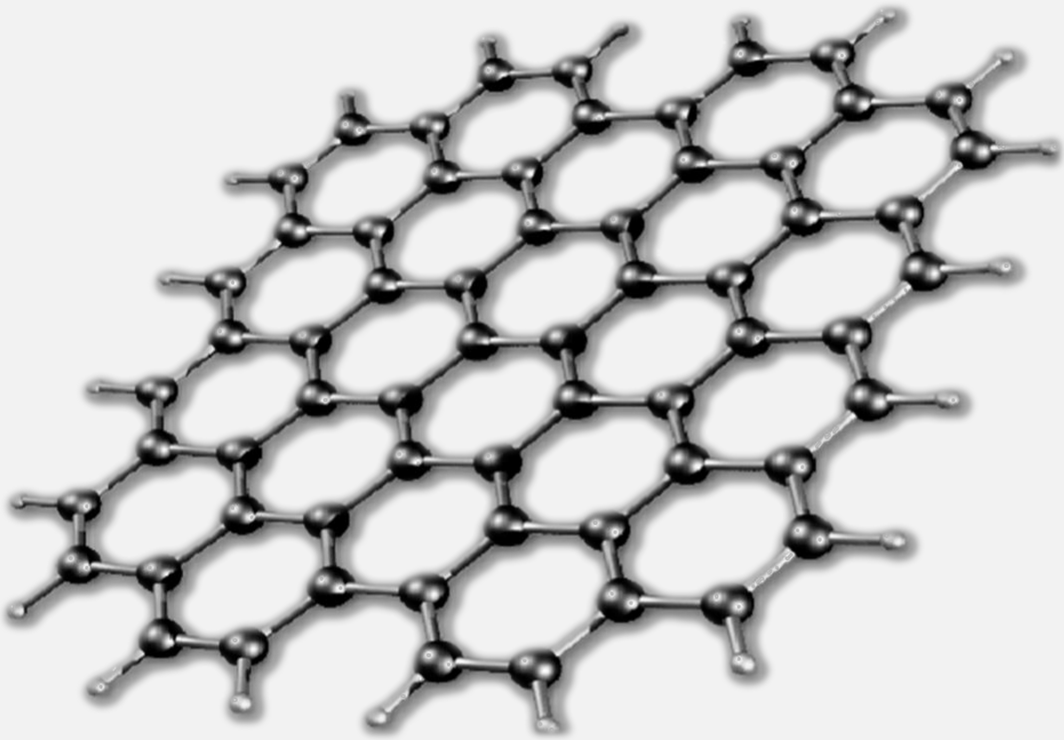
MISSION REPORT

« GRAPHENE SYNTHESIS »



Los Alamos National Laboratory

Center for Integrated Nanotechnologies

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ACKNOWLEDGMENT

First of all, I want to thank my tutor Enkeleda Dervishi, as well as CESI who gave me the chance to do this internship. I would also like to thank my colleague Justin Marquez who trained me in the use of scientific equipment and who very quickly enabled me to become autonomous in my work.

Finally, I would like to thank my company Revima APU for allowing me to do my internship abroad during these four months, and to discover new ways of working, thinking and living in the United States.

SUMMARY

INTRODUCTION

This report describes the activities I carried out during my internship at the Center for Integrated Nanotechnologies (CINT) of the Los Alamos National Laboratory (LANL).

I will begin by setting the context in which this internship took place. I would then talk about the differences between American and French cultures and their impact on daily life and working methods.

Finally, I will describe what graphene is, its applications, the different ways to synthesize it, characterize it, then transfer it to the desired substrates.

Graphene has many possible applications (gas detection, fuel cells, photovoltaic cameras, bionanoapplications, etc.).

I have worked on graphene which is a material that has been growing rapidly in recent years. The main reason for this boom is the interest of its physical and mechanical properties.

My mission was to understand graphene synthesis and learn how to characterize this material via various instruments.

I. INTERNSHIP CONTEXT

A. USA

The United States of America (USA) was founded in 1776, when the first 13 colonies were established on the Atlantic coast. Today, it is a federation of fifty states. Alaska and Hawaii were the last two states included in 1959. The Capital City is Washington D.C.

Figure 1 USA Flag [1]

The United States is the third largest country in the world (behind Russia and Canada). There are about 2800 miles (4500 km) between the east coast and the west coast. 1550 miles (2500 km) separates the northern border (Canada) and the southern border (Mexico).

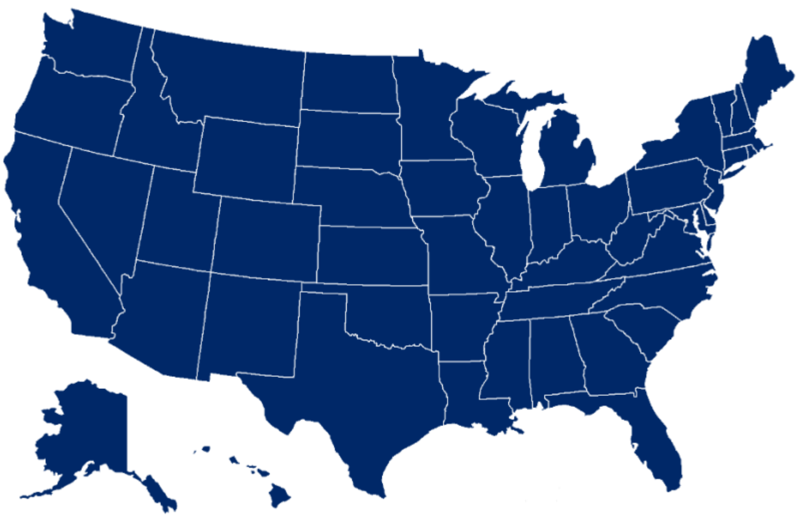

Figure 2 Map of the USA [1]

The population is about 300 million. It is the third most populous country in the world (after China and India).

The country is divided into four regions: the Northeast, Midwest, South and West. These regions differ in size, population, race, dialects and cultural origin.

B. NEW MEXICO

New Mexico is located in the southwestern United States. It is bordered to the west by Arizona, to the north by Colorado, to the east by Texas and to the south by Mexico. New Mexico has an area of 314,926 km^2 and is populated by approximately 2 million people. Its shape is close to a 550 km^2. The Capital City of the state is Santa Fe.

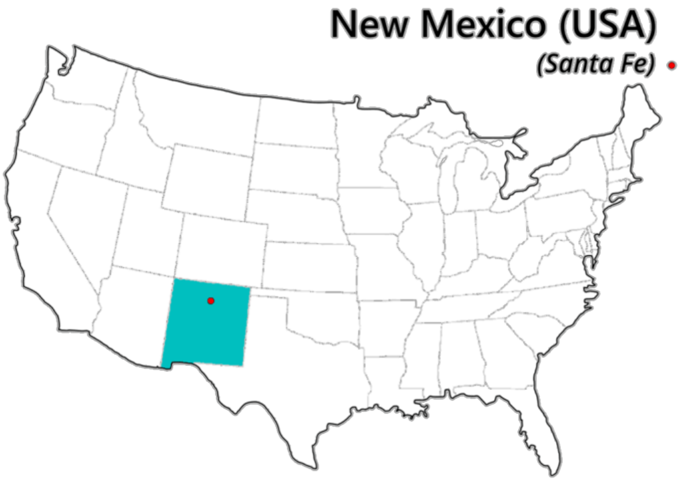

Figure 3 Location of New Mexico [2]

New Mexico's climate is semi-arid, although there are areas of mountainous climates. The territory is mainly composed of mountains, high desert plains and deserts. There is a large number of reliefs called "Mesa" as well as canyons formed by local rivers such as the famous Rio Grande.

Figure 4 Picture of the Rio Grande near Abiquiu Lake

C. LOS ALAMOS

Los Alamos County is a small community that possesses a world-wide reputation for scientific and technological development. Cultural and recreational opportunities abound, with plenty of hiking and biking trails, skiing at the local Pajarito Ski Resort, and visits to nearby Valles Caldera National Preserve and Bandelier National Monument.

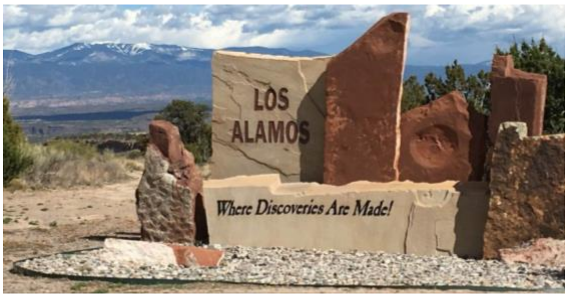

Figure 5 Los Alamos entrance panel [4]

Los Alamos is located on the Pajarito Plateau, in the mountains of Northern New Mexico, at 7,355 feet altitude (2242 m). It is approximately 90 miles north of Albuquerque, 35 miles from Santa Fe, and 55 miles from Taos. Los Alamos is surrounded by national forest, national park, pueblos and other federal lands. It is the smallest county in New Mexico, covering 109 square miles.

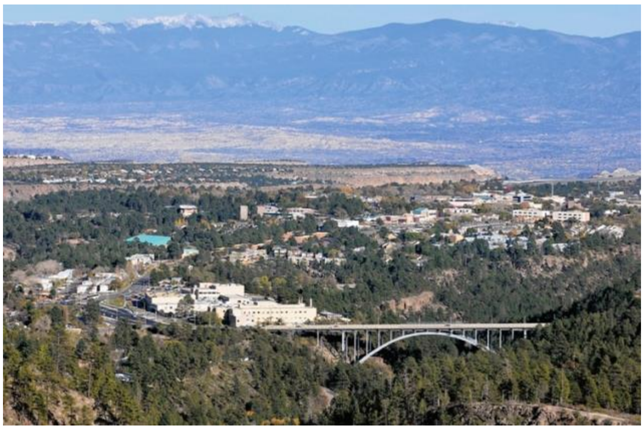

Figure 6 View of Los Alamos [4]

The population of Los Alamos is approximately 17,000 people. There are two communities in the County: the townsite of Los Alamos has about 10,500 residents and another 6,500 residents live in the community of White Rock, a few miles southeast of Los Alamos. Los Alamos National Laboratory is the largest employer in the County. Approximately 7,000 people commute to work at the Laboratory - traveling to Los Alamos from Northern New Mexico, Santa Fe and the Albuquerque metro area, and nearly doubling the Los Alamos population during a standard work week.

D. LOS ALAMOS NATIONAL LABORATORY

The laboratory was established in 1943 in Los Alamos for the Manhattan project to design and build the first atomic bomb. On July 16, 1945, the first atomic bomb exploded 200 miles south of Los Alamos. This project was then under the direction of General Leslie Groves, while the direction of the scientific staff was managed by Robert Oppenheimer.

Figure 7 LANL gate in 1953 [5]

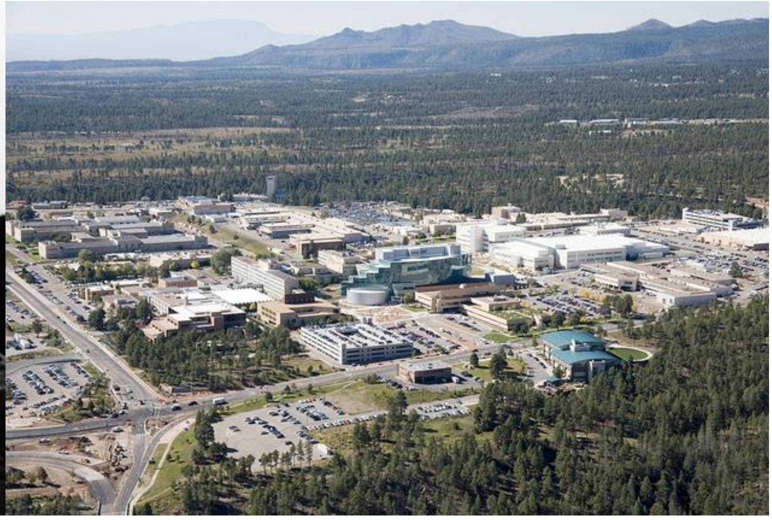

Figure 8 View of LANL today [6]

Today, the laboratory always keeps in mind the safety, security and reliability of the country's nuclear deterrence. However, LANL has also opened up to other fields of study such as:

- Physics
- High-performance computing
- Energies
- Biology
- Chemistry
- Earth and environmental science

LANL has also focused on raising workers' awareness of safety and security, with the core values of intellectual freedom, scientific excellence and national service still present. All this with the aim of working on nuclear non-proliferation and border security, energy and infrastructure security, as well as measures to combat nuclear and biological terrorist threats.

E. CENTER FOR INTEGRATED NANOTECHNOLOGIES

Figure 9 CINT Logo [7]

As a part of LANL, the Center for Integrated Nanotechnologies is a Department of Energy-funded nanoscience research facility. The CINT operates as a national user facility devoted to the design, performance, and integration of nanoscale materials. The CINT provides open access to tools and expertise needed to explore the continuum from scientific discovery to the integration of nanostructures into the micro and macro world.

Figure 10 CINT [7]

II. AMERICAN CULTURE

The American culture is different on many points: communication, behavior, work organization, daily life, etc. I was mainly in Los Alamos when I was at work, however I was living in Santa Fe, the capital of New Mexico. So my observations are based on the 2 main cities where I spent time.

A. ORDERS OF MAGNITUDE

Unlike France (and Europe in general) who mandated the use metric units in 1824, the United States uses imperial units. They only use the metric system for scientific purposes. All the orders of magnitude that we habitually use in France must be converted to be able to be understood. For example, kilograms in ounces or pounds, degrees Celsius in degrees Fahrenheit, liters in gallons and especially lengths in inches, feet and miles.

In the United States, everything from cars to food is huge. For example, the roads are very wide because the cars are bigger than in Europe (half of them are pick-ups). Gasoline is also cheaper ($2/gallon (0.45 €/L)) so the purchase of large cars is not a problem. For food, this can range from drinks that can be bought per gallon (1 gallon = 3.8 L) to hamburger sizes XXL. The urban and rural landscapes are also gigantic, skyscrapers can be seen in every major city, the distances to travel from one city to another are also impressive.

B. ENVIRONMENT

I live in Normandy where there are fields of greenery as far as the eye can see and where the climate is very cold and humid. So it was a shock for me to arrive in a region as hot and dry as New Mexico. Santa Fe and Los Alamos are located at about 7000 feet altitude (2000 m), so the air is poorer in oxygen, so it is very difficult to breathe there the first days. In addition, there are risks of dehydration and sunburn due to dry air and less filtered UV rays at high altitudes.

Regarding the wildlife, it's more threatening with tarantulas, rattlesnakes, mountain lions or brown bears. However, the landscapes are very beautiful. Los Alamos is a city on a mesa, in the middle of a rocky desert, while Santa Fe is located on a high plain from where we can observe the surrounding mountains.

C. BEHAVIOR

1. EVERYDAY BEHAVIOR

I was immediately warmly welcomed by all the Americans I met. They introduce themselves to each new person they meet and start a direct discussion to learn more about you. They are not afraid to tell details of their lives from the first topics of discussion. I also noticed that they love introducing you to new people with a brief description of yourself, which allows you to quickly expand your circle of friends.

The English language, much easier to learn than French, facilitates discussions between people. Even if your English is not perfect, you can make yourself understood very simply, without being judged on the quality of your English.

2. PROFESSIONAL BEHAVIOR

a. Feedbacks

Americans give much more feedback on your work, whether it is positive or negative. For example, I was entitled to "good jobs" whenever I managed to do a task independently. It helps a lot to have self-confidence when doing delicate tasks that we have never done in the past. The communication is direct with them, they do not hesitate to take you back or let you know if you have things to improve in your work method. Working in the laboratory, I was often given advice on how to handle chemicals better or to save time in the various processes to be used.

b. Professional hierarchy

In France, we can easily recognize the position of someone by his clothes or how you talk to him. In USA, it’s not true, the professional hierarchy is less formal than in France. Americans, at all hierarchical levels, are called by their first name or nickname, which makes it easier to approach their superiors. Manager and supervisor are not necessarily dressed in suit, they dress normally to not show that they are higher than you in the hierarchy. They always take time to come in your office to know if everything is good and are always present if you need their help.

c. Documentation

The documentations are more precise than in France, they want to be sure that you understand all the processes you will use in your work and all the details you should know about safety or security. The access to the various laboratories and chemical products is given only after extensive training specific to each laboratory (like safety gas training, chemical wastes, etc.).

III. PRESENTATION OF THE STUDY

A. WHAT IS GRAPHENE ?

Graphene was discovered in 2004 in England by two scientists. Graphene sheets are simply thin slices of graphite crystals (material from which the pencil leads are made). It is composed of carbon atoms assembled into a hexagonal "honeycomb" structure.

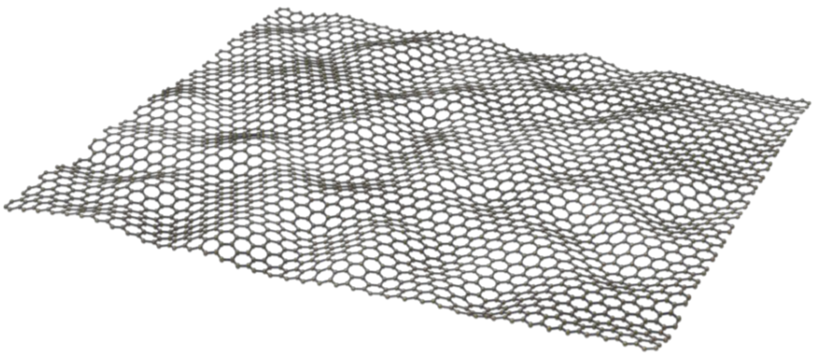

Figure 11 Graphene sheet [8]

Its tiny size (no larger than the size of an atom) and its high electron transfer speed allow all miniaturizations of electronic components, in order to replace the silicon that makes up almost all the electronic circuits that surround us. Flexible, transparent, and nearly 200 times stronger than steel for a weight six times less.

Here some examples of graphene applications are:

- Computer processors
- Waterproof coating
- Thin film solar panels
- Electrically conductive ink
- Infrared-vision contact lenses
- Medical diagnostic instruments
- Advanced pressure sensors
- Flat and flexible touch screens
- Quantum computer
- Batteries and supercapacitors

But its unique structure remains problems for production. To achieve the above performance, graphene must have perfect purity. There are different methods to produce graphene and during my internship at CINT, I learned one of them.

B. CHEMICAL VAPOR DEPOSITION

The process to produce graphene starts with a chemical vapor deposition (CVD) on a sheet of copper foil, heated to 1000°C in an inert atmosphere, into which methane (CH_4) gas is injected. The high temperature dissociates the methane into carbon and hydrogen atoms, and the carbon atoms settle onto the copper foil.

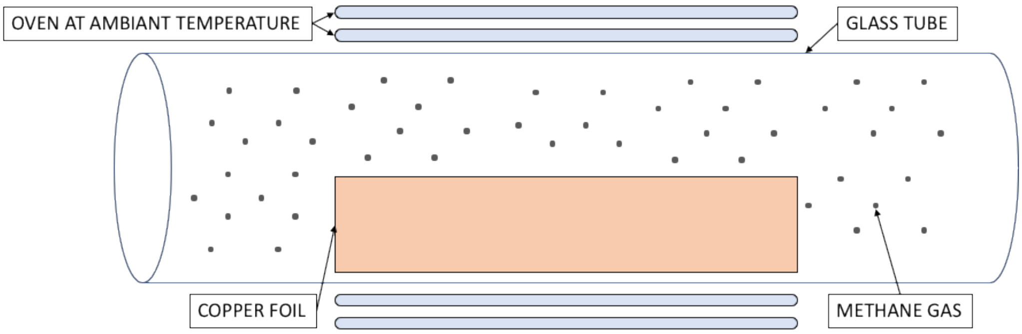

Figure 12 CVD before heating

Heat treatment, also called annealing, modifies the surface morphology of copper. Inert gasses (argon, and hydrogen) are used to dilute the oxygen present. Carbon not only goes to the surface of the graphene, but adsorbs to the surface. This occurs after the CH_4 molecule dissociates due to the extreme temperature of its growing environment (>1000C). Temperature provides enough energy to break the bonds of methane and allow carbon atoms to form a more stable network with itself (graphene), which reduces its entropy and becomes one of the most stable molecular structures known to man (hence its amazing physical properties: strength/weight ratio and conductance).

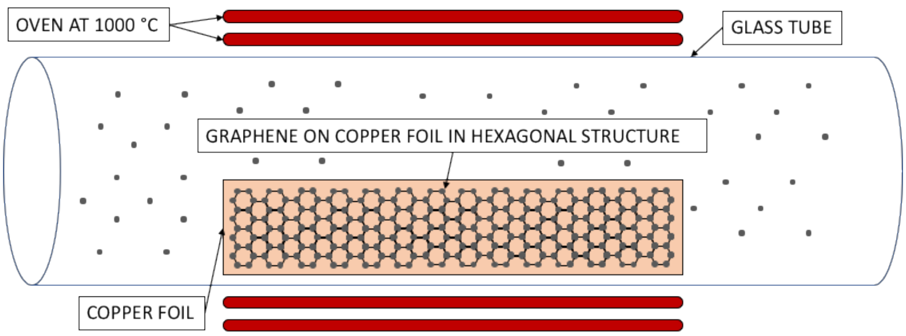

Figure 13 CVD during heating

At this point, however, it's not just graphene; graphene is attached to copper foil via Van Der Waals Forces. In chemistry, a van der Waals force is an electrical interaction of low intensity between two atoms, molecules, or between a molecule and a crystal. For example, one can find the effects of this force at the end of the legs of the gecko, thus ensuring their strong adherence on glass.

C. GRAPHENE CHARACTERIZATION

1. OPTICAL MICROSCOPY

The optical microscope is a type of microscope that uses visible light and a lens system to enlarge images of small samples. It is used to see the general appearance of the graphene layer. This allows us to have a first observation of the sample after the CVD.

Here are two samples of optical microscope images of copper foils before and after graphene growth:

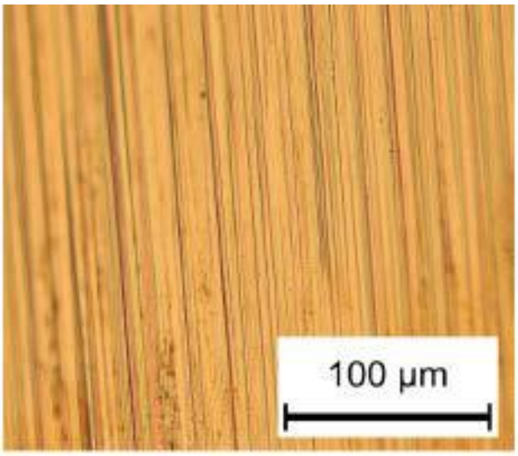

Figure 14 Copper foil before graphene growth [9]

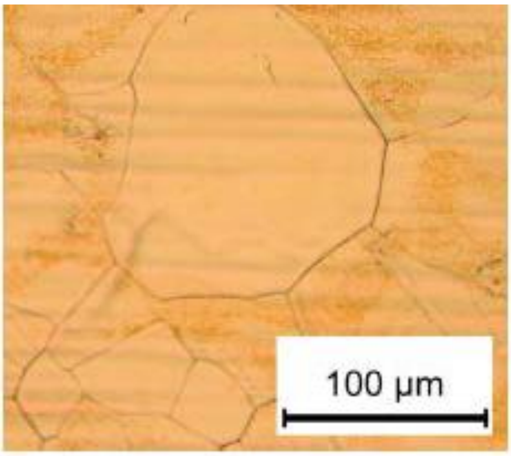

Figure 15 Copper foil after graphene growth [9]

2. RAMAN SPECTROSCOPY

We use Raman spectroscopy to determine the quality of a graphene layer because it is a nondestructive and fast technique to characterize carbon nanomaterials. Many graphene properties can be probed by the Raman spectrum, making it an ideal way to probe graphene without changing its properties.

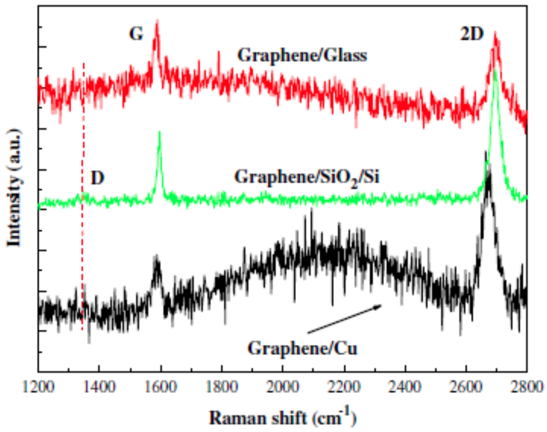

Figure 16 Raman spectra of graphene on various substrates [9]

The relative intensity of the ratio of the 2D and G peaks increases with the decrease in the number of layers. For example, a 2D/G ratio greater than 1 means that there is only one graphene layer. As for the ratio D/G, if it is less than 1, it indicates a very good specimen because of the low intensity of the peak D. To summarize, here is the information provided by each peak:

- 2D - Vibrational interaction
- G - sp2 carbon ordered/hybridized
- D - defects/disorder/entropy

Generally, the positions of these peaks are the following:

- 2D – 2700 cm^{-1}
- G – 1580 cm^{-1}
- D – 1350 cm^{-1}

A theory on the intensity of the D and G peaks is still under investigation, but it can be assumed that the higher the number of defects, the higher the intensity of the D peak.

The Raman spectra of cultivated graphene and graphene transferred to various substrates are compared, as shown in figure 16. Strong G and 2D peaks are detected for graphene transferred to silicon dioxide/silicon (SiO_2/Si). Similarly, G and 2D peaks are observed, but no D peaks in the Raman spectra of cultured graphene transferred to glass, indicating excellent graphene crystallinity. Graphene transferred to polished silicon shows a better signal because it can reflect more light without scattering, leading to a better signal from the detector. Using the same method one should get the exact same transfer quality regardless of substrate.

3. SCANNING ELECTRON MICROSCOPY

We use another type of microscope, the scanning electron microscope (SEM), which produces images of a sample by scanning the surface with a focused electron beam.

By scanning the sample surface with high-energy electrons and monitoring the secondary electrons backscattered from the surface, a very clear image of the sample surface can be obtained.

In our case, SEM is used to study the microstructure of graphene:

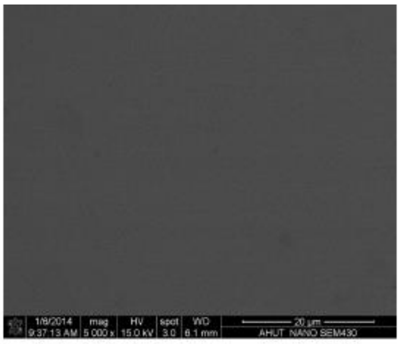

Figure 17 SEM image of graphene [9]

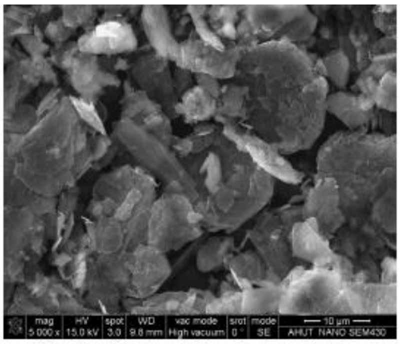

Figure 18 SEM image of graphite powder [9]

As shown in the SEM images above, graphene has a very smooth surface, which indicates its high quality, compared to graphite powder which is composed of crystals of different sizes. These results also confirm the large difference in microstructure between graphene and graphite.

4. ELECTRON BACKSCATTER DIFFRACTION

Because the quality of the copper foil we use has a great influence on the quality of the cultivated graphene, we also use electron backscatter diffraction (EBSD). This is a crystallographic micro structured electron microscopy method, where the sample is visualized by electrons backscattered by the sample surface. EBSD is used to study the evolution of the crystal structure of a metal film, and to characterize the growth of different crystallographic orientations.

To simplify, with this method, we can observe all the microscopic imperfections present on our copper sheet.

Here is an illustration of a copper foil observed via EBSD, indicating the crystalline orientations of the observed sample, using a color corresponding to the angle of the crystal direction.

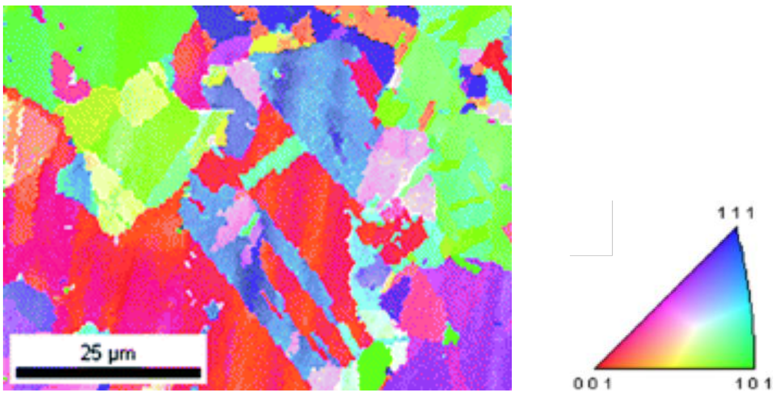

Figure 19 EBSD image of copper foil [10]

D. GRAPHENE TRANSFER AND POST GROWTH PROCESSING

Once the graphene is grown on the copper foil, the objective is to transfer it to the device that will be used for its application. For that, we must succeed the transfer without damaging too much the graphene structure. There are 3 different transfer methods.

1. THERMAL RELEASE TAPE

The first step is to apply the thermal release tape (TRT) to the graphene, and press it to a specific pressure level. The copper is immersed in acid for several hours. When the copper is dissolved, we remove the acid from the TRT and place it on the substrate. The final step is to heat the TRT and remove it so that only graphene remains.

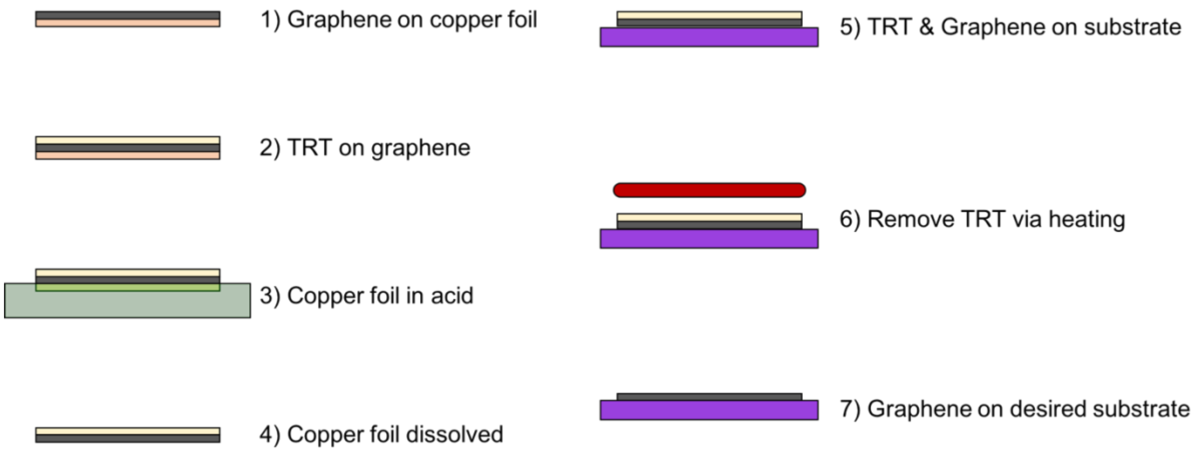

Figure 20 Graphene transfer via TRT

2. PMMA

The first step of the process consists in depositing an aqueous solution of polymethylmethacrylate (PMMA) on the graphene grown by CVD. The whole is then passed to the centrifuge in order to allow PMMA to properly hook the graphene. After the copper is dissolved, the PMMA/graphene block is rinsed with deionized water and transferred to the substrate. PMMA is then dissolved with acetone.

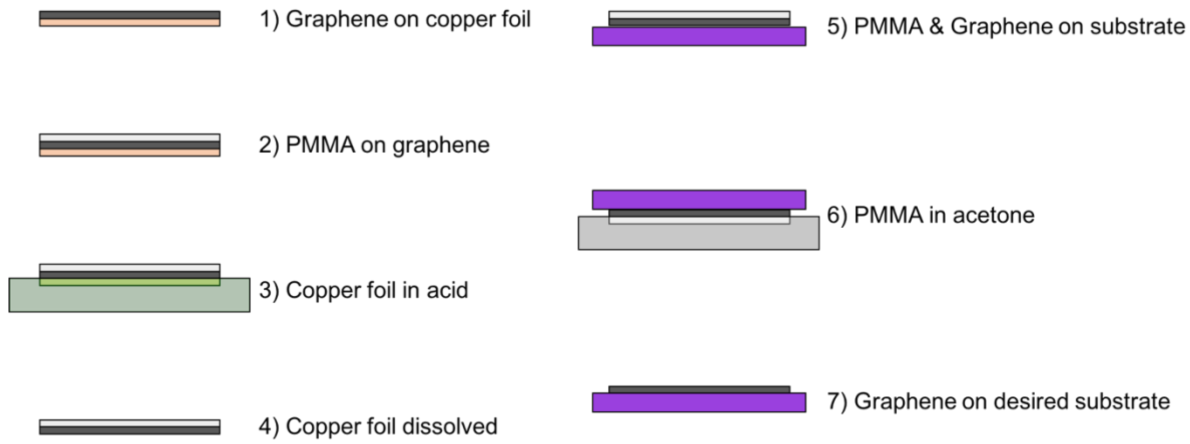

Figure 21 Graphene transfer via PMMA

3. FORMVAR

The graphene and copper foil are immersed in liquid formvar (polyvinyl formal). The formvar then dries in the open air and sticks to the graphene. The copper is then dissolved in acid. The formvar and graphene are then transferred onto the substrate. The formvar is then dissolved in chloroform.

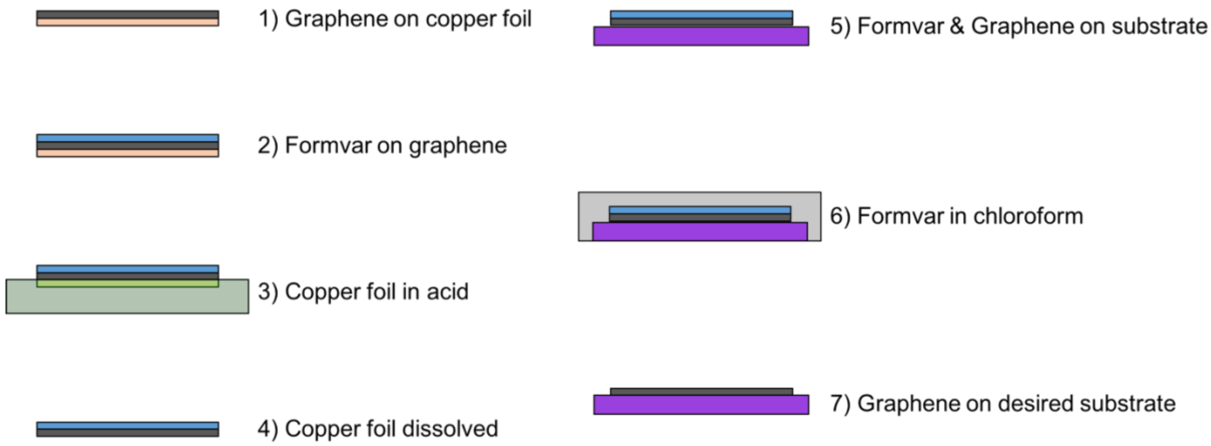

Figure 22 Graphene transfer via Formvar

4. COMPARATIVE METHODS

Unfortunately, PMMA and TRT leave a residue on the graphene surface (although the PMMA residue is considered tolerable for a particular application). These methods are also too expensive and time consuming to be used in production. That is why we are working on the formvar method.

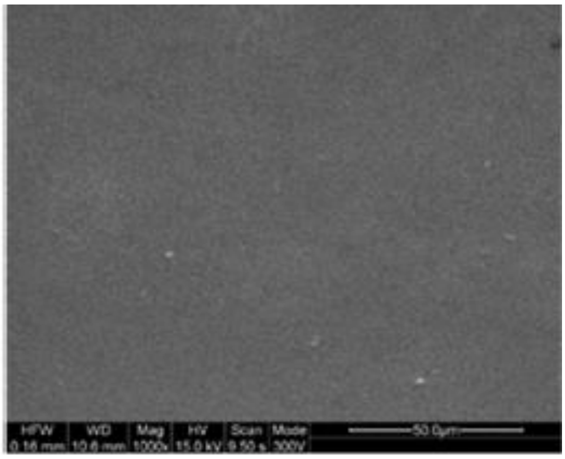

Figure 23 SEM of dissolution of formvar after 1 min [11]

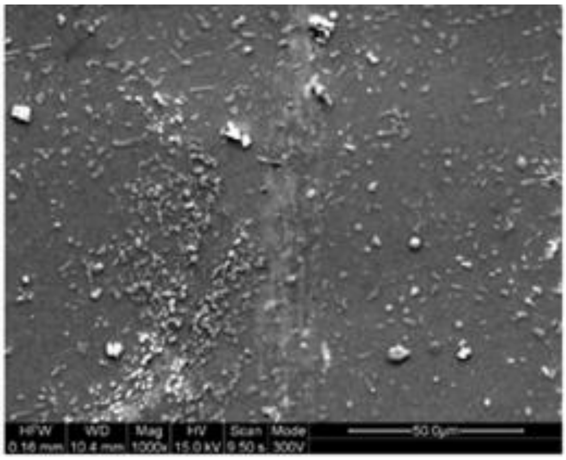

Figure 24 SEM of dissolution of PMMA after 1 min [11]

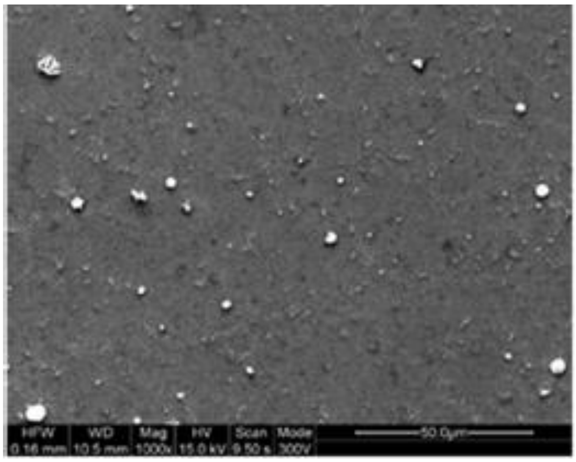

Figure 25 SEM of dissolution of PMMA after 60 min [11]

SEM images above show a sample of formvar/graphene/silicon after 1 minute in a chloroform solution. We notice that for the same time spent in acetone, the PMMA/graphene/silicon sample does not have the same quality at all, and those even when left exposed after 60 minutes.

CONCLUSION

This was my first lab experiment. I was able to observe important differences between the world of research and the world of business. The methodology is totally different, in the laboratory everything must be controlled and recorded in order to accurately use every piece of information that can influence the results of experiments. The equipment made available is also much more detailed and must therefore be used with great care, so I had to provide a lot of training work before I could perform experiments in the laboratory.

We don't have much opportunity to speak English during classes in France. Thanks to this internship in the USA, I was able to speak English with a lot of people, whether at work or when I went out on weekends. I was also able to work on my oral comprehension thanks to the different accents that people can have here. In France, we are always afraid of what people think about us so it’s difficult for us to try to speak English because we are afraid of making mistakes. An immersion in the USA helped so much, 3 or 4 weeks after my arrival, I was able to speak easily with people and my English was more fluent. People don’t judge you if you make mistakes as long as you try to speak with them.

Living in a foreign country alone helps you to be autonomous and resourceful. If you don’t have a car, you have to find a way to go to work every day or shop for groceries. You also have to be less shy and take upon yourself to speak to people if you want to make friends, even if American people are easy to approach.

Finally, this immersion in the American way of life allowed me to see beyond French way of life. It helps you to see assets you have in France and don’t have in the USA like a good social security, inexpensive school, and good quality food. But you can also see which kind of problems they haven’t here like the price of gasoline, traffic jam, and low salary.

GLOSSARY

USA	*United States of America*
NM	*New Mexico*
LANL	*Los Alamos National Laboratory*
CINT	*Center for Integrated Nanotechnologies*
CVD	*Chemical Vapor Deposition*
SEM	*Scanning Electron Microscope*
EBSD	*Electron Backscatter Diffraction*
TRT	*Thermal Release Tape*
PMMA	*Polymethylmethacrylate*
FV	*Formvar*

REFERENCES

[1] United States, From Wikipedia, the free encyclopedia:
https://en.wikipedia.org/wiki/United_States

[2] New Mexico, From Wikipedia, the free encyclopedia:
https://en.wikipedia.org/wiki/New_Mexico

[3] Los Alamos National Laboratory website:
http://www.lanl.gov

[4] Los Alamos County website:
https://www.losalamosnm.us/

[5] Manhattan Project Photo Galleries
https://www.energy.gov/management/photos/manhattan-project-photo-galleries

[6] Keeping Los Alamos safe, secure, and environmentally responsible
http://www.lanl.gov/discover/publications/connections/2017-06/science.php

[7] Center for Integrated Nanotechnologies website:
http://cint.lanl.gov/

[8] Graphene
https://en.chem-station.com/gallery/2014/01/graphene.html

[9] Jincheng Fan, Tengfei Li, Yuanhong Gao, Jianguo Wang, Hanlin Ding, Hang Heng: *Comprehensive study of graphene grown by chemical vapor deposition*. New York. Springer Science & Business Media. 2014.

[10] Jong-Kwon Lee, Chang-Soo Park, Heetae Kim:
Sheet resistance variation of graphene grown on annealed and mechanically polished Cu films.
http://pubs.rsc.org/en/content/articlehtml/2014/ra/c4ra11734d

[11] Eric Auchter, Justin Marquez, Stephen L. Yarbro, Enkeleda Dervishi:
A facile alternative technique for large-area graphene transfer via sacrificial polymer.
https://aip.scitation.org/doi/10.1063/1.4986780

ILLUSTRATION TABLE

1 Rue G. Marconi
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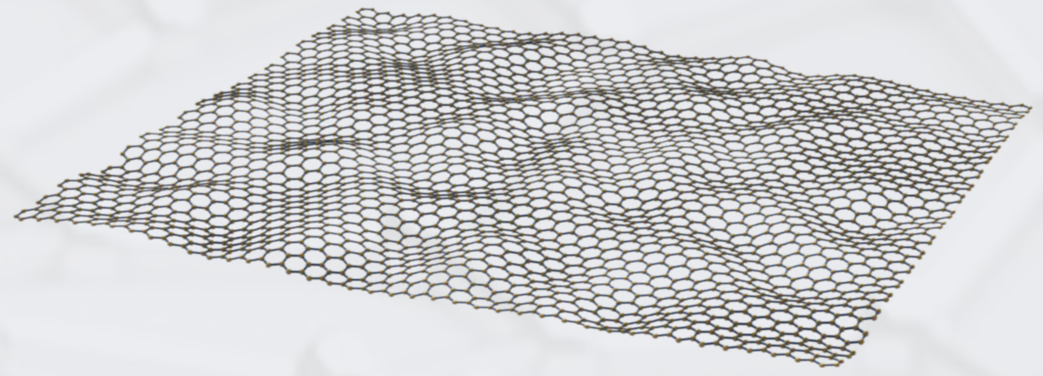
Center for Integrated Nanotechnology
Los Alamos National Laboratory
Los Alamos, NM, USA

WHAT IS GRAPHENE ?

Graphene is composed of thin slices of graphite crystals (material from which the pencil leads are made). It is composed of carbon atoms assembled into a hexagonal “honeycomb” structure.



Graphene sheet

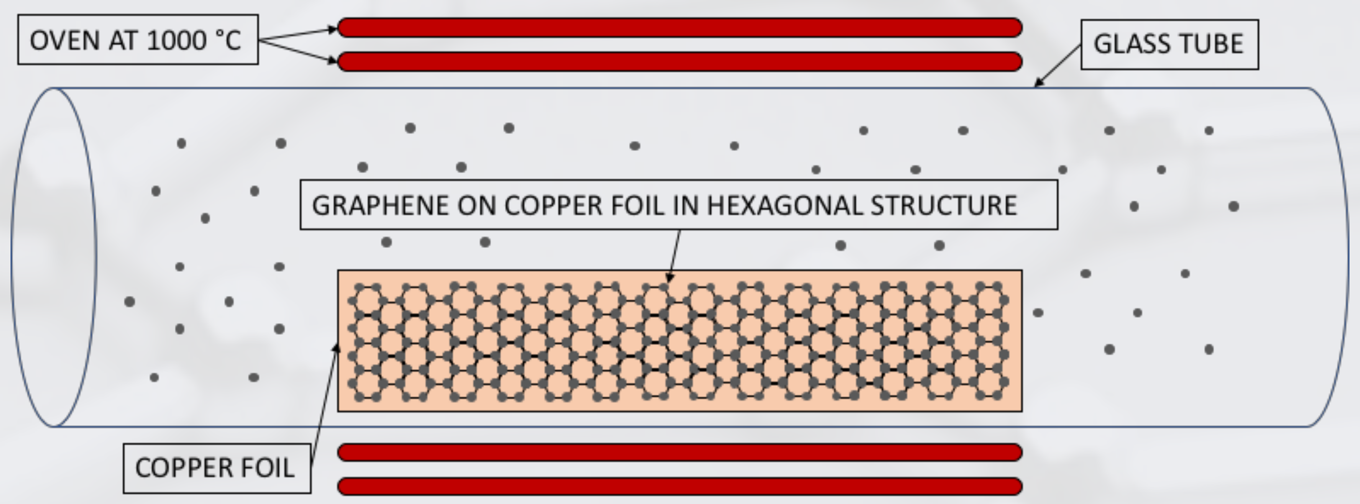
One slice is no larger than an atom. Graphene has high electron transfer speed that allow miniaturizations of electronic components. Flexible and transparent, it also has a resistance to breakage nearly 200 times greater than steel for a weight six times less important.

But Its unique structure remains problems for production. To achieve the above performance, graphene must have perfect purity. There are different methods to produce graphene and it is the a one of the missions of the CINT to test these different methods.

CHEMICAL VAPOR DEPOSITION

This is the process used to growth graphene:

1. Copper foil with a high purity (around 99%) is put into a glass tube.
2. This glass tube is then vacuumed to be sure that there is no more oxygen.
3. Argon and hydrogen are injected in the tube to dilute the oxygen present so that it does not ruin the graphene.
4. Then, the glass tube is heated with an oven at approximately 1000 °C.
5. When the tube and the copper foil are at the desired temperature, methane is injected in the tube.
6. Next cool down.
7. Then the copper foil is retrieved with the grown graphene.

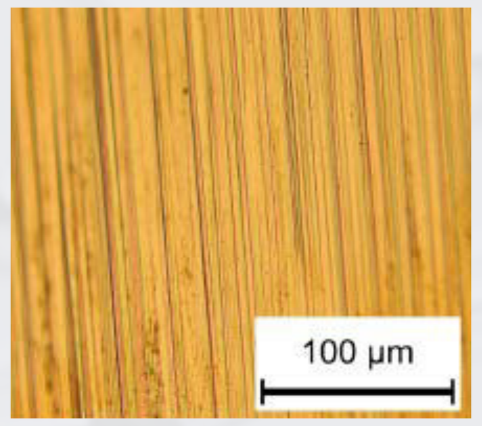


Growth of graphene during CVD

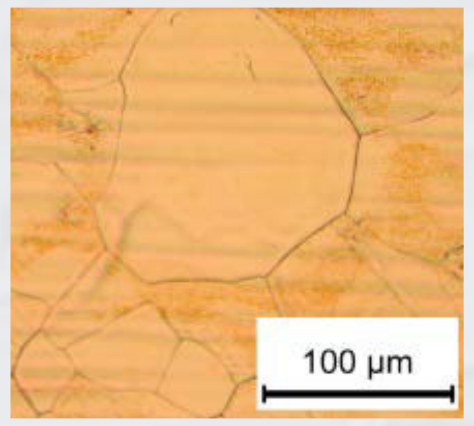
Heat treatment, also called annealing, modifies the surface morphology of copper. Carbon not only goes to the surface of the graphene, but adsorbs to the surface. This occurs after the CH_4 molecule dissociates due to the extreme temperature of its growing environment. Temperature provides enough energy to break the bonds of methane and allow carbon atoms to form a more stable network with itself (graphene).

GRAPHENE CHARACTERIZATION

Optical Microscopy:
The optical microscope is a type of microscope that uses visible light and a lens system to enlarge images of small samples. It is used to see the general appearance of the graphene layer. This allows us to have a first observation of the sample after the CVD.

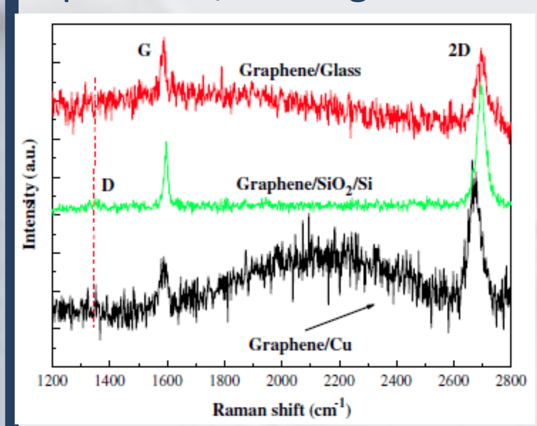


Copper foil before graphene growth



Copper foil after graphene growth

Raman Spectroscopy:
Raman spectroscopy is used to determine the quality of a graphene layer because it is a nondestructive and fast technique to characterize carbon nanomaterials. Many graphene properties can be probed by the Raman spectrum, making it an ideal way to probe graphene without changing its properties.

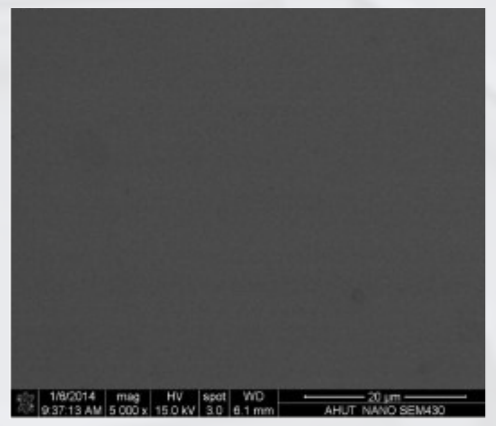


The relative intensity of the ratio of the 2D and G peaks increases with the decrease in the number of layers. For example, a 2D/G ratio greater than 1 means that there is only one graphene layer. As for the ratio D/G, if it is less than 1, it indicates a very good specimen because of the low intensity of the peak D.

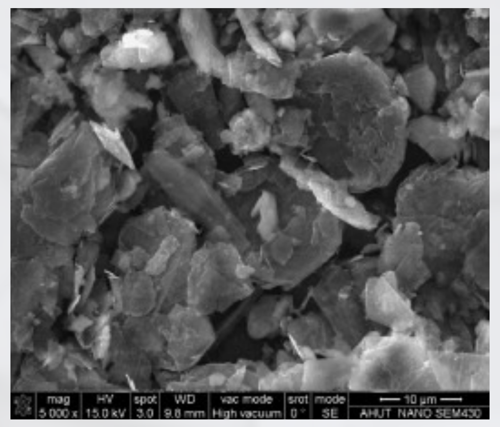
To summarize, here is the information provided by each peak:

- 2D - Vibrational interaction
- G - sp2 carbon ordered/hybridized
- D - defects/disorder/entropy

Scanning Electron Microscopy:
An other type of microscope is used, the scanning electron microscope (SEM), which produces images of a sample by scanning the surface with a focused electron beam. By scanning the sample surface with high-energy electrons and monitoring the secondary electrons backscattered from the surface, a very clear image of the sample surface can be obtained.

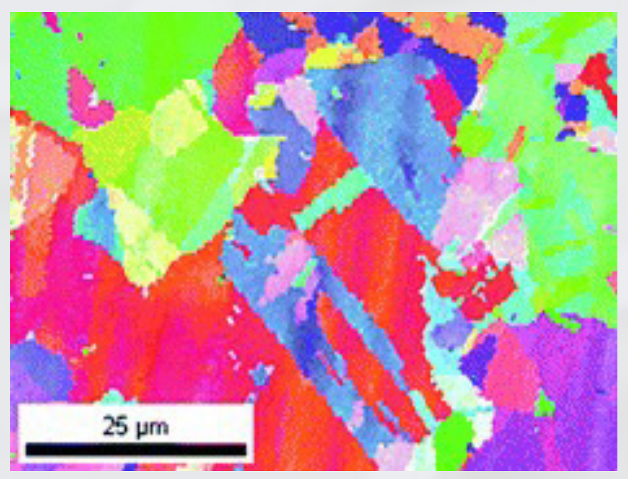


SEM image of graphene



SEM image of graphite powder

Electron Backscatter Diffraction:



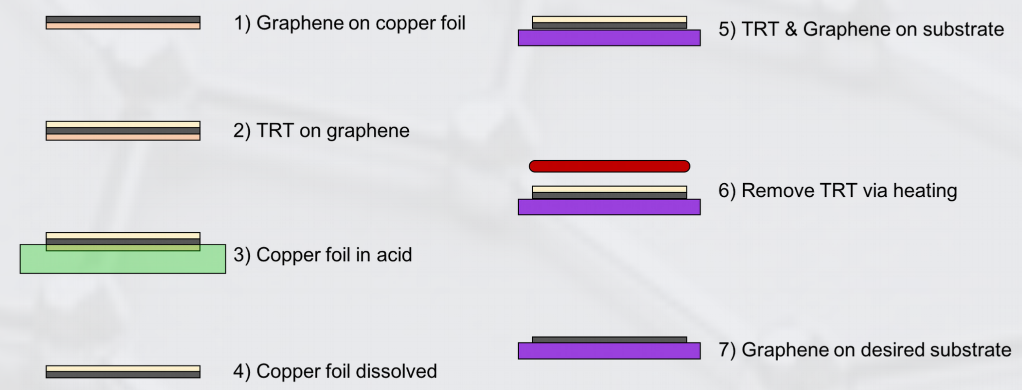
EBSD image of copper foil

Because the quality of the copper foil used has a great influence on the quality of the cultivated graphene, the electron backscatter diffraction (EBSD)is also used. This is a crystallographic micro structured electron microscopy method, where the sample is visualized by electrons backscattered by the sample surface. To simplify, with this method, all the microscopic imperfections present on our copper sheet can be observed using a color corresponding to the angle of the crystal direction.

TRANSFER OF GRAPHENE

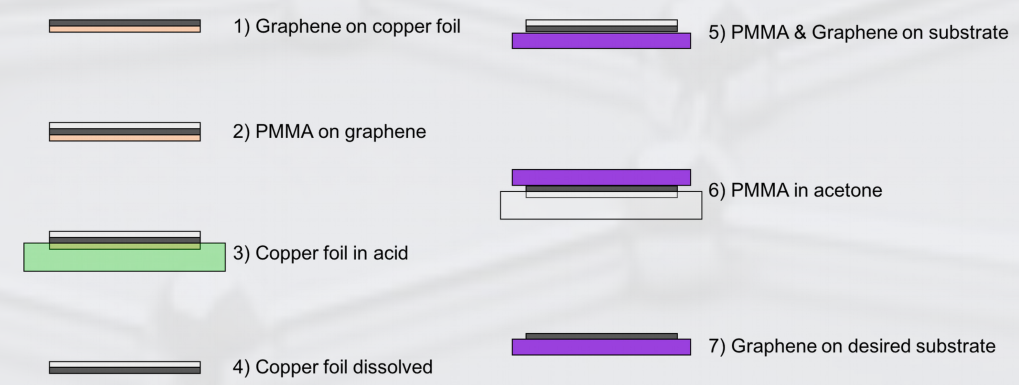
Once the graphene is grown on the copper foil, the objective is to transfer it to the device that will be used for its application. For that, the transfer must be succeed without damaging too much the graphene structure. There are 3 different transfer methods.

Thermal Release Tape:
The first step is to apply the thermal release tape (TRT) to the graphene, and press it to a specific pressure level. The copper is immersed in acid for several hours. When the copper is dissolved, the acid is removed from the TRT and placed on the substrate. The final step is to heat the TRT and remove it so that only graphene remains.



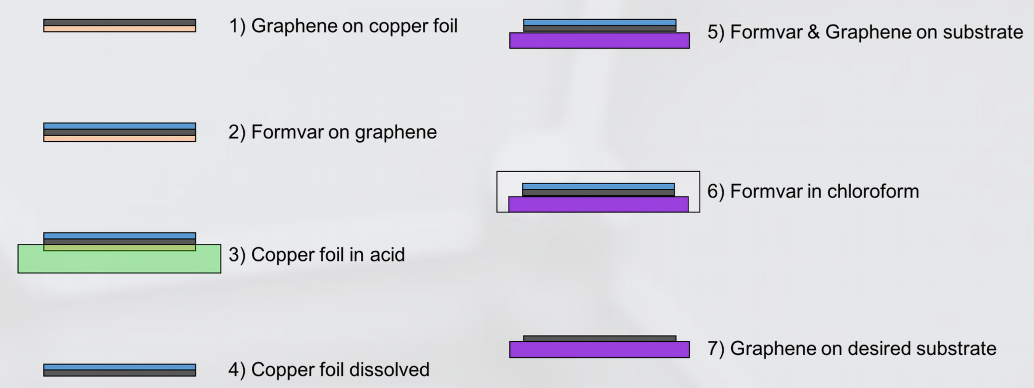
Graphene transfer via TRT

PMMA:
The first step of the process consists in depositing an aqueous solution of polymethylmethacrylate (PMMA) on the graphene grown by CVD. The whole is then passed to the centrifuge in order to allow PMMA to properly hook the graphene. After the copper is dissolved, the PMMA/graphene block is rinsed with deionized water and transferred to the substrate. PMMA is then dissolved with acetone.



Graphene transfer via PMMA

Formvar:
The graphene and copper foil are immersed in liquid formvar (polyvinyl formal). The formvar then dries in the open air and sticks to the graphene. The copper is then dissolved in acid. The formvar and graphene are then transferred onto the substrate. The formvar is then dissolved in chloroform.



Graphene transfer via Formvar

References:

- Graphene, https://en.chem-station.com/gallery/2014/01/graphene.html
- Jincheng Fan, Tengfei Li, Yuanhong Gao, Jianguo Wang, Hanlin Ding, Hang Heng: *Comprehensive study of graphene grown by chemical vapor deposition*. New York. Springer Science & Business Media. 2014.
- Jong-Kwon Lee, Chang-Soo Park, Heetae Kim: *Sheet resistance variation of graphene grown on annealed and mechanically polished Cu films*. http://pubs.rsc.org/en/content/articlehtml/2014/ra/c4ra11734d
- Eric Auchter, Justin Marquez, Stephen L. Yarbro, Enkeleda Dervishi: *A facile alternative technique for large-area graphene transfer via sacrificial polymer*. https://aip.scitation.org/doi/10.1063/1.4986780